

# News Briefs

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## General Developments

*Inquiries about News Briefs, where no contact person is identified, should be referred to the Managing Editor, Journal of Research, National Institute of Standards and Technology, Building 416, Room 119, Gaithersburg, MD 20899-0001; telephone: 301/975-3572.*

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### ATP SETS SIX NEW FOCUS AREAS FOR 1995

Six new focused areas of technology—all based on input from U.S. industry—have been defined by NIST's Advanced Technology Program under which companies and consortia will be able to propose projects for \$785 million of long-range, cost-shared support. The new focused technology programs are founded on ideas from "white papers" submitted by industry and workshops held around the country. Projects selected for funding will be cost-shared and carried out by industry. This announcement does not constitute a call for proposals at this time; competition deadlines for each of the six focused areas will be set in the near future. The programs, all slated for five years, are: Motor Vehicle Manufacturing Technology, \$185 million; Catalysis and Biocatalysis Technologies, \$160 million; Materials Processing for Heavy Manufacturing, \$145 million; Digital Data Storage, \$125 million; Digital Video in Information Networks, \$120 million; and Advanced Vapor Compression Refrigeration Systems, \$50 million. For summaries of the new focused program areas, fax a request to (301) 926-1630 or send an e-mail message to: baum@micf.nist.gov (via Internet). This information also is available electronically from the NIST gopher (gopher-server.nist.gov). For information on the ATP, call (800) ATP-FUND (287-3863) or send e-mail to: atp@micf.nist.gov (via Internet).

### RUSSIA, NIS EXPERTS OFFERED U.S. STANDARDS TRAINING

To enhance trade between the United States, Russia and the Newly Independent States (NIS) of the former Soviet Union, NIST and the American National Standards Institute are cooperating with the International Trade Administration's Special American Business Internship program, or SABIT, to develop a comprehensive standards-related training program for qualified engineers, administrators, and technical and regulatory experts from Russia and the NIS. The aim of the SABIT program is to provide technical training and contacts in support of both commercial and technical cooperation between the United States and the NIS to improve international trade. Training for 100 interns will be conducted in 1995 and will focus on five proposed target industries: automotive, medical equipment, telecommunications, infrastructure, and aerospace. For information, contact Mary Saunders, Office of Standards Services, TB417 Rm. 106, NIST, Gaithersburg, MD 20899-0001, (301) 975-2396, fax: (301) 963-2871.

### UNITED STATES, INDONESIA TO HARMONIZE STANDARDS

To enhance trade between the United States and the Republic of Indonesia, the U.S. Department of Commerce and the Indonesian Ministry of State for Research and Technology have signed a memorandum of understanding on scientific and technical cooperation to remove non-tariff trade barriers between the two countries. The MOU was signed on Nov. 14, 1994, in Jakarta by U.S. Commerce Secretary Ronald H. Brown and Indonesian Minister of State for Research and Technology B. J. Habibie. The agreement designates NIST and the Indonesian National Standardization Council

to implement a common set of activities under the umbrella of the MOU. The agreement recognizes the growing importance of the harmonization of standards and conformity assessment measures to improve international trade. Indonesia is included in a category of countries identified by the Commerce Department as "Big Emerging Markets." These are countries that hold promise for large incremental gains in U.S. exports. For information, contact the Office of International and Academic Affairs, A505 Administration Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-3089, fax: (301) 975-3530, e-mail: OIAA@micf.nist.gov (via Internet).

#### **GROUP SEEKS IMPROVED SEMICONDUCTOR COMPETITIVENESS**

NIST's Manufacturing Extension Partnership (MEP), in cooperation with another Department of Commerce agency, the Bureau of Export Administration, and the Department of Energy's National Laboratories System, has established a consortium with the goal of reducing the manufacturing costs and improving the worldwide marketability of I/O pins. The pilot effort hopes to strengthen the competitive position of the U.S. semiconductor industry. I/O pins are the component of an integrated circuit that provides the avenue for electric current to pass from the semiconductor chip to the circuit board. In the new NIST-led consortium, pin manufacturers and semiconductor package suppliers will seek ways to improve energy efficiency, pin cleaning and finishing methods, manufacturing processes, sources of long-term financing, technical training, cooperative buying methods and supplier relations. Companies who join the consortium also are asked to sign cooperative research and development agreements with NIST. For more information on the U.S. Semiconductor Pins Pilot Project, contact Aaron Leventhal, MEP, Polymer B115, NIST, Gaithersburg, MD 20899-0001, (301) 975-4510, e-mail: aaron@enh.nist.gov (via Internet).

#### **OPEN SYSTEMS WORKSHOP PLANS FOUR 1995 SESSIONS**

The Open Systems Environment Implementors' Workshop will convene quarterly in 1995 to work toward agreements on information technology standards for open systems environments. The 1995 meetings will take place on March 14–16, June 13–15, Sept. 12–14 and Dec. 5–7. The meetings are sponsored by NIST and the Institute of

Electrical and Electronics Engineers Computer Society, and will be held at NIST headquarters in Gaithersburg, MD. For more information, contact the OSE Implementors' Workshop secretariat, NIST, B226 Technology Building, Gaithersburg, MD 20899-0001, (301) 975-3664, e-mail: bgray@sst.ncsl.nist.gov (via Internet).

#### **NANOSTRUCTURED MATERIALS WORKSHOP REPORT AVAILABLE**

A report from a recent workshop on a new class of high-technology materials with grain sizes or some other characteristic dimension on the order of a few nanometers will be of interest to researchers in industry, universities, and government. Fabrication control of these materials occurs at the nanometer level. They are a distinct class of materials with unusual properties that potentially have important industrial applications. Among several conclusions, workshop participants stated that a great deal more basic research is necessary in order to fulfill the industrial potential of these materials. NIST can provide assistance as an interface between industrial applications and basic research by conducting key missing-property investigations and modelling, preparing standards, assembling and administering a nanostructured materials information database, and making available to outside users such national characterization facilities as the NIST Cold Neutron Research Facility for the synthesis and study of these materials. For a copy of the report, First NIST Workshop on Nanostructured Materials, send a self-addressed mailing label to Robert D. Shull, B152 Materials Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-6035.

#### **ENERGY-RELATED INVENTIONS PROGRAM MAKES RECOMMENDATIONS**

During September and October 1994, the NIST Office of Technology Evaluation and Assessment recommended innovative electric vehicle, agricultural, and construction technologies to its DOE partner for commercialization support. These included:

- **Flywheel Electric Battery**—This is a flywheel system for energy storage particularly for use in electric vehicles and uninterruptable power supplies. The flywheel system consists of a flywheel supported by a set of patented magnetic bearings in an evacuated containment system. This particular flywheel system is the closest to a market-ready state of a number of technically

feasible competing designs, each with good commercial and economic potential.

- **Stalk and Root Embedding Apparatus**—This device buries agricultural debris, e.g., stalks and roots, and is particularly suited for row crops such as cotton. The invention enables farmers to meet regulatory requirements to plow under post-harvest debris to stop pest growth while reducing cost and energy requirements of this tillage. The device operates behind a tractor to embed debris in a single pass. Following its use, the field is ready for replanting.
- **Power Guard**—The invention is the integration of thin-film amorphous-silicon photovoltaic (PV) cell modules into roofing material for use on flat or low-slope roofs of commercial buildings. The system is to be marketed to building owners and investors in selected electric utility areas as a method for reducing peak power demand. Commercial feasibility is enhanced through state and federal energy tax credits and utility rate structures that encourage on-peak PV power generation.
- **Morris Modular Agricultural Carbonizer/ Incinerator**—This device burns agricultural waste from livestock production and dairy farming. Designed for farm site use, the heat energy is employed to sterilize and recycle water for animal use. Its widespread use, especially in the Midwest, e.g., the eight-state Ogallala aquifer region, can reduce the rapidly growing demand for water placed on large aquifers by increased livestock production. The device could have significant potential as an export product to underdeveloped countries. Field testing is to be accomplished by the Agricultural Engineering Group, Kansas State University.
- **Method of Constructing Insulated Foam Homes**—This is a patented construction method for a concrete post-and-beam structure encased in expanded polystyrene (EPS). The EPS panels create a highly insulated thermal envelop for a residential dwelling. An overall R-50 value for the exterior envelope is claimed for the EPS black/cast concrete wall and a truss-type roof framing system.

#### **SNS JUNCTION BREAKTHROUGH FOR ARRAY STANDARDS**

A NIST scientist has demonstrated an important breakthrough in PdAu-barrier superconductor-normal-superconductor (SNS) junctions for incor-

poration in voltage array standards. The scientist fabricated a test chip having junction-to-junction variations in critical current of less than 5 % in a 400-junction series array; precision voltage measurements confirmed that all junctions worked as designed. The division is pursuing a number of different junction technologies for the next generation of programmable voltage standards as part of its Josephson Array Development Project. SNS junctions are one candidate; these have specific advantages over tunnel junctions: larger critical currents, smaller size, and smaller capacitance. For voltage standards, these features translate into improved stability against both noise and chaos, and the potential to reach 10 V in a single-chip design.

A major challenge has been to achieve sufficiently uniform critical currents in the very small junctions. In tests with microwave energy applied at 5 GHz, the current-voltage characteristic exhibited constant voltage steps of 1.5 mA, about 100 times the amplitude of steps in the present dc Josephson standard. The uniformity was so good that the series array looked like a single junction with a first step at 4.14 mV—400 times the value for one junction. The degree of junction uniformity achieved shows that NIST has a process available for making arrays of 1.7  $\mu\text{m}$  diameter SNS junctions and 1.0  $\mu\text{m}$  diameter contacts.

The characteristics of these junctions closely match the requirements of the programmable voltage standard proposed by a NIST scientist. Compared to programmable devices now excited at 75 GHz, the lower operating frequency is more convenient, and should provide a resolution 15 times better. Further, the junctions are about 130 times smaller than those in the present voltage standard and do not require shunting resistors. The junctions' decreased size should allow a 10 V programmable standard to be fabricated on a single 1 cm by 1 cm chip. The next challenge is a big one: the present design of the 10 V programmable standard requires 1 million SNS junctions having sufficient uniformity and an acceptable yield of good junctions.

#### **SAMPLING COMPARATOR SYSTEM ERRORS REDUCED**

NIST scientists have developed a method of phase-plane compensation to reduce the nonlinearities of the NIST Sampling Comparator System (SCS). The SCS is a precision, equivalent-time, automatic waveform sampling system used to measure repetitive rf signals (having fundamental frequencies

from 1 MHz to 200 MHz), with rms accuracy comparable to that of thermal converters. The high rms accuracy is achieved in part through the use of a custom-designed ASIC (application-specific integrated circuit) strobed analog comparator developed by NIST.

However, due to small nonlinearities in the electronic circuitry of the SCS, distortion is a limitation in terms of the fidelity in measuring test waveforms. In response to requests from the instrumentation and automatic test equipment industry, which uses sampling comparator circuits in their products/systems, NIST collaborated with Sandia National Laboratories to investigate the use of a phase-plane compensation method for reducing the NIST SCS nonlinearities.

Phase-plane compensation tables describe non-linearity errors as a function of the signal's instantaneous value and its time-derivative. A setup was assembled to acquire and to process all the required data for performing phase-plane compensation, and a table generated at one frequency (by using a nearly pure sinusoidal source) was used to correct the SCS's non-linearity errors at other input signal frequencies. Investigators report that a table generated at 100 MHz has been shown to reduce the harmonic distortion of the SCS by as much as 20 dB for signal frequencies ranging from 1 MHz or less up to 100 MHz, with an increase in minimum effective bits (a measure of the ratio of signal-to-noise plus distortion) from 6 to 9.

#### **GROWING IMPORTANCE OF POLARIZATION MEASUREMENTS REFLECTED IN OPTICAL FIBER MEASUREMENTS SYMPOSIUM**

The increasing importance of polarization measurements to the optical fiber industry resulted in more than one-fifth of the program of the recent NIST-hosted Symposium on Optical Fiber Measurements being devoted to this subject. Held at NIST's Boulder Laboratories, the three-day symposium covered measurements on related components and systems as well as on optical fibers themselves and drew some 200 metrologists from 15 countries. Optical time-domain reflectometry and related technology and the characterization of optical fiber amplifiers were two other major areas addressed by the 10 invited and 40 contributed talks; time-domain reflectometry has been important in the symposium agenda since the first meeting in 1980.

Now recognized as a major international conference, the symposium was founded by NIST scien-

tists. NIST has sponsored the symposium from the beginning, with the cooperation of the Institute of Electrical and Electronics Engineers and the Optical Society of America.

#### **NIST, UNIVERSITY OF MARYLAND ESTABLISH A COLLABORATION TO DEVELOP AND CALIBRATE ATOMIC STEP HEIGHTS**

In a recent SEMATECH-motivated survey of semiconductor industry needs, NIST identified an emerging requirement for calibrated step heights of 1 nm or less. Currently, no method exists by which to fabricate stable standards of this height. NIST scientists and a professor of physics at the University of Maryland have established a collaboration to develop and calibrate atomic step heights. The professor's research involves the production of atomic steps using annealing techniques in ultra high vacuum and the identification of these steps by scanned probe microscopy.

The NIST calibrated atomic force microscope will be used for the calibration. The z axis of this microscope contains a capacitance sensor capable of 0.01 nm resolution. The professor's group at the University of Maryland will support fabrication of the standards by providing equipment, personnel, and expertise. A Ph.D. student in material engineering will be working full time on this project over the next several years. If successful, this project will extend the NIST step height calibration facility to the atomic limit.

#### **ISO MEETING ON MACHINE TOOLS CO-SPONSORED BY NIST**

NIST co-sponsored an ISO TC39/SC2 meeting Oct. 24-28, 1994, with the Association for Manufacturing Technology (AMT). ISO TC39/SC2 is responsible for developing general test conditions and methodologies for machine tools and specific implementations of these methods to various types of machine tools such as machining centers and grinding machines, etc.

ISO standards on machine tools are centered about the testing of individual elements of machine tools instead of a performance-based test as is the case of the U.S. standard. As a result, a considerable amount of time is usually spent discussing the allowable tolerances for individual elements of a machine tool. By participating in these ISO meetings, NIST aims to make sure that U.S. machine tool builders can meet those tolerances and to try and change the standards to ones based on machine tool performance.

International standards are used for buying and selling equipment between countries. U.S. participation in the development of these standards is very important from the point of view of U.S. industry. Lack of conformance to these standards will create significant obstacles to selling U.S. equipment abroad. European countries and Japan are involved heavily in these standard committees, influencing the outcome to their advantage. Through their participation in this meeting, NIST researchers were able to influence the committee to include statements consistent with the U.S. national standard for machining center testing.

#### **CAALS DEVELOPS A COMMUNICATION SPECIFICATION FOR INSTRUMENT-TO-CONTROLLER MESSAGING**

The Consortium on Automated Analytical Laboratory Systems (CAALS) has completed the initial version of its CAALS-I Communication Specification, which will be issued as a NIST Internal Report. This document fully describes the communication rules (protocol) and message format (syntax) for instrument-to-controller communication. CAALS-I is designed to provide platform-independent guaranteed message delivery and connectivity across a variety of physical links.

Currently, IEEE-488.1, EIA-232E, and Windows DDE (Dynamic Data Exchange) links are supported. CAALS-I makes maximal use of existing standards and provides a similar application programming interface across computing platforms that is independent of the physical link used. The consortium has developed demonstration implementations of the specification on computing hardware typically used with laboratory instrumentation—graphical user interface-based PCs and embedded microcontrollers—and a PC-based testing program that verifies the operation of instrument-end implementations. A CAALS-I implementor's guide and a generic ANSI C code kernel containing the protocol stack are in progress. The intent is to make it as easy as possible for instrument manufacturers to utilize and adopt the CAALS-I specification. CAALS documentation that has been released to the public is available on the CAALS anonymous ftp site ([caals.nist.gov](http://caals.nist.gov)).

#### **NEW PROGRAM ESTABLISHED WITH AIGER TO SUPPORT "CLEAN CARS"**

The new vehicle emissions imposed by the Clean Air Act of 1990 will require vehicle manufacturers to develop more sophisticated analytical tools to

demonstrate compliance. To address these new requirements, the Environmental Research Council has established an umbrella cooperative research and development agreement among the U.S. vehicle manufacturers, the California Air Resources Board, and the U.S. Environmental Protection Agency to identify and address critical technologies that will require improvement from their current state of the art. On Aug. 30, 1994 the American Industry/Government Emissions Research (AIGER) organization, formed as a result of this CRADA, met with NIST scientists to discuss new measurement technology and standards needs associated with the development of next-generation "clean" U.S. automotive vehicles.

Brief overviews of NIST ongoing research efforts and capabilities in flow measurement, microsensor array technology, infrared and microwave spectroscopic sensing technology, and on-demand generation of gas standards were given followed by discussion. Based on the presentations and subsequent discussions, upon AIGER's request, NIST agreed to establish a new program directed toward the development and critical evaluation of technologies for the real-time measurement of several targeted chemical species in automotive exhaust. FY 1995 activities will be directed toward efforts in exhaust flow measurement technology and standards, microsensor array technology for gas phase species, Doppler-free infrared and microwave spectroscopic measurement technology, and SRMs for oxygenated hydrocarbons in air.

#### **NIST HOSTS WORKSHOP FOCUSED ON QA FOR VITAMIN MEASUREMENTS**

A NIST quality assurance (QA) workshop was held Oct. 15, 1994 in Arlington, VA, for laboratories that measure vitamins in human serum. This workshop, held annually for the past 10 years, is part of a QA program supported by the National Cancer Institute (NCI) to study the possible cancer preventive benefits of vitamins in human populations at high risk of developing certain forms of cancer. As a result of this program, the measurement capabilities of participating laboratories for the determination of vitamins such as A, E, C, and carotenes have improved substantially. Increased accuracy has been achieved by providing proficiency test sera tri-annually; making available a Standard Reference Material with certified values of selected vitamins; and providing laboratory measurement training sessions, site visits, and consultation services. The QA program initially supported a

core of approximately 10 NCI-grantee laboratories. The program has since expanded to include more than 30 domestic and over 20 foreign laboratories. NIST assessment of the intra- and inter-laboratory measurement capabilities is used to help provide valuable clinical and epidemiological data regarding cancer chemopreventive efficacy of the constituents.

In addition to the laboratories involved in cancer chemoprevention studies, this program is beginning to attract interest in response to requirements imposed by the Clinical Improvement Amendments (CLIA), which extended federal regulation to all laboratories performing diagnostic testing. The program also will be expanded to include a vitamins in foods component to assist the U.S. food industry and regulatory agencies in accurate nutritional labeling and the public in making informed nutritional choices.

#### **JAPAN AND UNITED STATES BEGIN COMPARISON OF TEMPERATURE SCALES**

NIST and its Japanese counterpart, the National Research Laboratory of Metrology (NRLM), have begun a collaborative effort to assure the equivalence of their realizations of the International Temperature Scale of 1990 (ITS-90) in the low-temperature region. The collaboration is part of Japan's Twin Institutes Program, through which Japan funds exchange visits of NIST and NRLM scientists for 1 or 2 months.

Using the NIST low-temperature facility for realization of the ITS-90, a NIST scientist and a NRLM scientist compared realizations of the ITS-90 in the region from 1.25 K to 5.0 K. In this region, the scale is defined by the  $^4\text{He}$  vapor-pressure/temperature relations. Helium vapor-pressure thermometry is performed with a temperature-controlled cell containing high-purity liquid helium in equilibrium with its vapor. High-accuracy pressure measurements are needed for vapor-pressure measurement; NIST employs a piston gauge. This measurement, along with the vapor-pressure/temperature equations provided in the ITS-90, defines the temperature of the cell. Two of NRLM's rhodium-iron resistance thermometers were placed in intimate thermal contact with the vapor-pressure cell. These thermometers had been calibrated previously in this range against NRLM's preliminary scale realization, and they serve as transfer standards representing that realization. They also had been calibrated against NRLM's wire scale, which NRLM currently uses as its standard and which is traceable to a  $^4\text{He}$  vapor-

pressure realization performed in a different system at NRLM 15 years ago.

The NIST realization agrees to within 0.5 mK with NIST's previously maintained wire scale, which is traceable to the original measurements defining the vapor-pressure/temperature relations of the ITS-90. Through the collaborative work, the NIST realization was found to agree to within 0.3 mK with NRLM's wire scale. Also, the NIST wire scale was shown to agree with the NRLM wire scale to within 0.3 mK. Primary realizations at NIST and NRLM, however, show disagreements as large as 7.5 mK. This is considerably greater than the 0.1 mK uncertainty obtainable with the systems used in both laboratories. This disagreement is under investigation, and new comparisons are planned after NRLM performs a new realization this year.

#### **MEASUREMENT UNCERTAINTY—1994 EDITION OF TN 1297**

The 1994 edition of NIST Technical Note 1297, Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, recently was distributed to the NIST technical staff. Additional copies of the 20-page publication may be obtained from the NIST Calibration Program, (301) 975-2002. The 1994 edition of TN 1297 includes a new appendix—Appendix D—that gives additional guidance on a number of topics related to measurement uncertainty, including the use of certain terms such as accuracy and precision. This document is expected to be even more useful than the 1993 edition, of which 10,000 copies were distributed worldwide. Readers wishing to explore the subject of measurement uncertainty in greater depth may purchase a copy of the ISO Guide to the Expression of Uncertainty in Measurement on which TN 1297 is based from ANSI.

#### **SOLDER JET PRINTING FOR MICROELECTRONICS APPLICATIONS**

The printing of solder patterns onto chips or circuit boards using ink jet printer technology is a novel means to avoid cumbersome screening techniques and environmentally unfriendly electroplating methods. In addition, "solder jet" technology is flexible (patterns can be input by keyboard) and capable of very fine pitch between solder deposits ( $<100\text{ }\mu\text{m}$ ). These capabilities are needed for the next generation of microelectronic packaging.

NIST is working with a private company to bring ink jet technology closer to commercial reality.

NIST scientists are focusing on materials compatibility issues involving the liquid solder and the jetting apparatus, and reactions between the jetted solder and the various substrate materials used in microelectronic interconnects. Working closely with the private company and the consortium members, they have identified means to overcome technical barriers to good mechanical and electrical contacts in these solder joints.

#### **METHOD DEVELOPED FOR PROCESS MONITORING OF THE POLYMER PRECURSORS FOR CERAMIC MATRIX COMPOSITES**

A method for monitoring the transformation of liquid prepolymers into solid polymer networks has been developed by scientists at NIST. The method will be used in a private company program sponsored by the Advanced Research Projects Agency to improve the manufacture of ceramic-matrix composite products by employing resin transfer molding (RTM) technology. The initial application will be ceramic components of aircraft engines. In RTM, liquid reactants are injected into a mold containing fiber reinforcements and heated to transform the liquid into a solid polymer. Pyrolysis of the solid polymer combined with reinfiltration of further liquid reactants produces the ceramic composite. Information about the completeness of the conversion of liquid resin into solid polymer is important for optimization of the manufacturing process.

The new monitoring method uses infrared spectroscopic surface sensing to monitor the chemical reactions in real time. Infrared spectroscopy is commonly used to measure the kinetics of chemical processes. The challenge is to adapt the instrumentation to make on-line measurements in the manufacturing environment. The developed technique uses flexible fibers that transmit infrared radiation to and from a prism mounted in the manufacturing mold and thereby achieves contact with the resin during processing. The returning infrared radiation is analyzed by conventional infrared instrumentation. The technique has been demonstrated in the NIST laboratories with the resins of interest. The information will be used by process control software also being developed by NIST to automate the fabrication process. Plans are under way to adapt the method to the manufacturing operation at the private company.

#### **POLYCRYSTALLINE $\text{Bi}_2\text{Te}_3$ THERMOELECTRIC ELEMENTS FOR REFRIGERATION**

$\text{Bi}_2\text{Te}_3$ -based alloys have excellent thermoelectric properties and commonly are used for refrigeration applications. They replace all conventional mechanical parts in a conventional refrigerator as well as the refrigerants that harm the atmosphere. To increase the robustness and ease of manufacturing of  $\text{Bi}_2\text{Te}_3$ -based elements, scientists at NIST have been studying different techniques to produce polycrystalline  $\text{Bi}_2\text{Te}_3$  elements with optimum thermoelectric properties. Presently, the lack of crystallographic preferred orientation (texture) decreases the thermoelectric efficiency of  $\text{Bi}_2\text{Te}_3$  making polycrystalline elements undesirable for commercial applications. To increase the texture of bulk samples, a new chemical co-precipitation route has been developed that yields platy sub-micrometer powder of  $\text{Bi}_2\text{Te}_3$ . The platy morphology of the particles allows a high degree of texture to be introduced in polycrystalline bulk samples made by hot-pressing and axisymmetric hot-forging. Research is still in progress to understand texture development and its effects on thermoelectric efficiency. Several alloys of Bi-Te-Se and Bi-Te-Sb-Se have been produced with promising thermoelectric properties.

#### **NIST DEVELOPS HELICOPTER PACKAGE TO MEASURE SMOKE FROM LARGE OUTDOOR FIRES**

NIST developed and tested a smoke measuring package designed to be suspended beneath a helicopter. The package, which was designed for the U.S. Coast Guard, was developed as part of the effort to examine the feasibility of intentional in situ burning of oil spills. The package provides real-time measurement of carbon dioxide and hydrocarbon concentrations in large smoke plumes and hydrocarbon emissions from oil spills. Concentration measurements from the package, along with time, altitude, and position data from a satellite global positioning system, temperature, barometric pressure, and wind speed are transmitted to a ground station via a radio modem and displayed on a laptop computer. The package also collects smoke samples, which are analyzed for particle size distribution and smoke yield.

The helicopter package was tested successfully in October at the U.S. Coast Guard's Fire and Safety

Test Detachment in Mobile, AL. The package was deployed from a Coast Guard helicopter to measure hydrocarbon emissions from a controlled diesel fuel spill in a 15 m square pan. The diesel fuel was ignited, and the package was used to make measurements in the smoke plume 300 m downwind from the fire.

A fixed wing aircraft from SRI International with an on-board LIDAR laser detection system was used to measure the distribution of smoke particulates in the smoke plume. These measurements will provide verification information for the large eddy simulation downwind smoke plume dispersion computer model under development at NIST.

#### **NIST ESTABLISHES CENTER FOR HIGH INTEGRITY SOFTWARE SYSTEMS ASSURANCE**

As a collaborative approach for government, industry, and academia to make available the technology necessary to assure high integrity software in a growing number of applications, NIST has established the Center for High Integrity Software Systems Assurance (CHISSA). The goal of the center is to facilitate the cooperation of researchers and developers in seeing how research results perform in practice, improving the dependability of the resulting applications, and directing research efforts toward helping developers with their real problems.

#### **NEW PUBLICATION PRESENTS RESULTS OF FLAT PANEL DISPLAYS (FPDS) WORKSHOP**

NIST Special Publication 500-219, NIST Workshop on the Computer Interface to Flat Panel Displays, reports on a workshop sponsored by NIST on Jan. 13–14, 1994, in San Jose, CA. The meeting was attended by about 55 people from more than 40 computer, FPD, and graphics controller companies as well as representatives from government, both civilian and military.

Objectives of the workshop included determining the need for a standard or series of standards for the computer interface to FPDs, defining what types of standards are needed, identifying approaches for developing FPD interface standards, and obtaining a consensus on a coordinated plan for standards development. Workshop attendees agreed on the need for a standard flat panel display interface for integrated devices and encouraged the participation of FPD manufacturers in the standards activities.

#### **NIST MEASURES FIELDS IN FULL-SCALE AIRCRAFT IN SHIELDING EFFECTIVENESS STUDY**

At the request of the General Aviation Manufacturer's Association, the Federal Aviation Administration has funded NIST to study the shielding effectiveness of aircraft skins and structure. The primary output of the NIST study will be an improved characterization of the actual fields within aircraft to determine appropriate test field levels and methods for electromagnetic immunity testing of aircraft electronics.

The aircraft industry currently faces a difficult testing challenge due to concerns regarding the susceptibility of aircraft electronics to high-intensity radiated fields (HIRFs). Even though the HIRF environment has been fairly well characterized, the shielding effectiveness of aircraft skins and structures is not well known. Consequently, there is no rigorous basis for specifying the levels of external fields applied to an aircraft for test purposes.

In the most recent investigation, NIST scientists conducted time-domain electromagnetic field measurements in a Boeing 707 airframe as a result of irradiating the aircraft with a horn antenna placed at several locations around the aircraft. The time-domain method isolated the effects of ground-reflected waves to provide a reference field that was more representative of free-space conditions. The time constant was measured directly for both the passenger cabin and the cockpit, and the quality factor ( $Q$ ) of the cabin and cockpit cavities was calculated from the time-constant data. Shielding effectiveness of the structure was measured for three different radiation angles of incidence; attenuation in the cockpit was found to be in the 10 dB to 20 dB range. These measurements followed measurements and calculations of shielding effectiveness, time constant, and  $Q$  made on three small twin-engine civilian aircraft.

The measurement results are in general agreement with theoretical predictions of an earlier model developed by the division for aperture penetration into lossy aircraft-structure-like cavities and with measurements made on a laboratory model of an aircraft cavity with apertures and internal loading. Further, the results show that the electromagnetic environment in aircraft cavities is well simulated by a reverberation chamber. Accounting for internal loading, i.e., absorption by lossy objects, such as people, is important because it affects both the steady-state field levels inside the aircraft and the time constant for the buildup of pulsed radar fields.



## **NIST INVESTIGATES PYTHON PROGRAMMING LANGUAGE**

NIST scientists have been evaluating the Python language for use in applications such as the development of graphical user interface applications, including "control panels" to manage interacting software applications used in the design of microwave tubes; probing models of the Very High Speed Integrated Circuit Hardware Description language; and an enhancement to the National Center for Supercomputing Applications' MOSAIC World Wide Web client. Python also is being considered by the Automated Electronic Manufacturing Project (AEMP) for use in developing application-testing demonstrations for the SEMATECH computer-integrated manufacturing framework specification.

Python is an interactive, object-oriented language intended for use by end users and available free over the Internet. To assist in their use of Python, NIST scientists invited the program's author, a staff member of the Netherlands research organization CWI, to NIST. After he arrived, the AEMP team decided to obtain input from U.S. industry users of Python on the changes and enhancements they would like to see made in the language. An Internet invitation resulted in some 20 individuals from industry (plus a few from NIST) attending a three-day workshop; one of the outcomes of the workshop was a recommendation to pursue the organization of a Python association, or consortium. Following the workshop, the program's author gave a lecture to more than 40 NIST attendees, who were asked if they were interested in tutorials. The response was enthusiastic, and to date, four half-day tutorials have been presented, each focusing on different aspects of the language.

## **NEW SYMPOSIUM DEVELOPED AT ASME INTERNATIONAL CONGRESS**

A NIST scientist was the co-organizer and co-chairman, along with a professor from the University of Oklahoma, of the Symposium on Measurement and Inspection of Products and Processes, held Nov. 8, 1994 at the American Society of Mechanical Engineers (ASME) International Mechanical Engineering Congress and Exposition in Chicago. The symposium was one of several organized by the Production Engineering Division of ASME.

The symposium addressed issues in manufacturing metrology, process measurement, and product control and was organized into sessions dealing with surface evaluation and product and process measurement. A NIST scientist also participated in the symposium as a session co-chairman and delivered a paper entitled "Stylus Flight in Surface Profiling." The papers have been published as part of the proceedings volume, Manufacturing Science and Engineering, 1994, Vol. 68-1. The scientist intends to propose holding the symposium on a biennial or triennial basis at future ASME annual conferences because the ASME provides an important audience for such a forum on metrology for manufacturing.

## **NIST/IEEE CO-SPONSORED SMART SENSOR WORKSHOP**

NIST co-sponsored a second workshop with the Institute of Electrical and Electronics Engineers on sensor interface standards on Sept. 19, 1994, in cooperation with the Sensors Expo '94 in Cleveland, Ohio. The workshop's objective was to test the feasibility of developing a hardware-independent communication interface standard for smart sensors and actuators. The workshop started with a presentation of the summary of the first workshop conducted on March 31, 1994 at NIST, followed by a review of the latest control and sensor networking technology. End users and system integrators from various industrial sectors expressed their views for a smart sensor interface network. Viewpoints on the feasibility of a hardware-independent connectivity to the sensor network were presented by workshop participants. An open forum was held on the creation of a smart sensor interface standard and was widely discussed. The workshop concluded with a proposal to form three working groups to pursue future sensor communication standard activities:

- Network Communication Standard;
- Analog Sensor to Microprocessor Communication Standard; and
- Market Study.

The workshop was highlighted by keynote speech on the "Proliferation of Control Networking and its Global Impact on the Coming Century's Applications."

### NEW SPECIAL TEST SERVICE APPROVED AT NIST

NIST has approved a new Special Test Service that will evaluate the performance of data analysis algorithms embedded in coordinate measuring systems. The service is called the Algorithm Testing and Evaluation Program for Coordinate Measuring Systems (ATEP-CMS) and was developed by NIST researchers. ATEP-CMS is the first calibration program test of software used in dimensional metrology.

In the ATEP-CMS service, tested software is treated as a filter that transforms point coordinate data into feature parameters according to a defined transfer function. NIST evaluates the accuracy of the filter under conditions typical of those found in industrial practice. NIST generates simulated data sets and compares the output of the software under test to corresponding reference values determined by NIST through independent means. The technical elements of ATEP-CMS have been developed in coordination with the drafting of a new standard, Methods for Performance Evaluation of Coordinate Measuring Systems Software, by the B89.4.10 Working Group of the American Society of Mechanical Engineers. NIST also has been working to harmonize these efforts with similar programs in Germany, the United Kingdom, and the International Organization for Standardization (ISO).

### CHEMICALLY SPECIFIC RESPONSE PATTERNS FROM TEMPERATURE-PROGRAMMED GAS SENSORS

NIST researchers have developed a new approach to solid-state gas sensing that uses micromachined devices to produce chemically specific responses to gases. A U.S. patent has been issued for the NIST microsensors. The rapid thermal response of these miniaturized devices can produce chemically specific time-dependent signal patterns.

The sensors were fabricated on a silicon chip by a conventional CMOS (complementary metal oxide semiconductor) foundry followed by an anisotropic silicon etch and deposition of the sensing film. The etch produced thermally isolated microbridges consisting of a polysilicon heater, an Al hot plate, and conducting electrical contact pads, separated from each other by SiO<sub>2</sub>. The sensing film consisted of Pd- or Pt-doped SnO<sub>2</sub>. The device can be heated to 500 °C in 1 ms. By applying a repetitive train of voltage pulses of different amplitudes to the heater, rapid changes in temperature and, therefore, in the surface chemistry of the sensing film could be

induced. These changes were detected by measuring the conductance of the sensing film between pulses. The conductance response pattern to a specific pulse train is unique to the gas being sensed. Using pattern recognition techniques, a smart sensor can interpret these dynamic response patterns to identify the gas. Chemically specific response patterns for CO, H<sub>2</sub>, and the vapors of ethanol, methanol, acetone, and formaldehyde have been determined. Example applications of this technology include environmental sensors, fugitive emission sensors for chemical plants, and automotive gas analyzers. Because of the low power consumption (1 mW to 50 mW) of these microsensors, portable solid-state gas sensors are now possible.

### NIST DELIVERS LEAK STANDARD AND CALIBRATION SYSTEM TO THE NAVY

NIST scientists designed, constructed, and delivered a primary leak standard and calibration system to the Navy Metrology Engineering Center. This system operates with almost any gas between 10<sup>-14</sup> mol/s and 10<sup>-6</sup> mol/s (10<sup>-10</sup> std cc/s and 10<sup>-2</sup> std cc/s) and will support a variety of Navy leak-testing needs.

The Navy must verify leak integrity for nuclear and chemical waste containers, as well as vacuum-sealed components such as gyroscopes and radar tubes. To meet environmental regulations and maintain a habitable environment in submarines, the Navy must also ensure that the leakage rate from sealed chemical systems (e.g., refrigerators) remains below specified levels. Commercial calibrated leaks are not available for all of the test gases, nor do they cover the entire needed leak range. The extended capabilities of the NIST system not only will fill these gaps but also will reduce the current Navy leak calibration costs by about \$150,000 a year—paying for the system in less than 3 years. Greater savings are expected when the Navy performs leak calibrations for the other services.

### CHARACTERIZATION OF HFCs AND SOLID ACID CATALYSTS BY PROMPT GAMMA-RAY ACTIVATION ANALYSIS

NIST researchers, in collaboration with chemists at a private company, have used cold neutron prompt gamma-ray activation analysis (PGAA) to study a number of materials of industrial importance. The instrument, located in the NIST Cold Neutron

Research Facility, uses a beam of low energy neutrons to induce emission of gamma-radiation in samples. The technique is well-suited to the non-destructive determination of hydrogen and other light elements.

In one such application, PGAA was used to study the molecular adsorption of hydrofluorocarbons (HFCs) on zeolites. HFCs, which are replacing ozone-destroying chlorofluorocarbons in a wide range of uses, may be purified by adsorption on zeolite molecular sieves. These sieves allow size-selective transport of chemical products and reagents, as molecules become trapped inside the zeolite supercage structure. PGAA was used to determine the H/Na ratio in samples of NaX zeolite ( $\text{Na}_{86}\text{Al}_{86}\text{Si}_{104}\text{O}_{384}$ ) containing adsorbed HFCs. The results indicated a total of four HFC molecules per zeolite supercage, providing strong evidence that the HFC molecules are bound to a site in the zeolite structure, which contains four Na ions.

In a second study, PGAA was used to characterize a class of catalysts known as solid superacids, which are important for chemical reactions such as isomerization of hydrocarbons. Certain metal oxides, such as  $\text{ZrO}_2$  and  $\text{TiO}_2$ , have been observed to behave as solid superacids when small amounts of sulfate and hydrogen are incorporated onto their surfaces. These solid acids may eventually replace liquid acids such as  $\text{H}_2\text{SO}_4$  and HF in industrially important processes, thereby reducing the waste streams generated. PGAA was used to study the stoichiometry of  $\text{ZrO}_2$  and  $\text{TiO}_2$  superacids as a function of calcining temperature and time. The researchers found that sulfur and hydrogen concentrations in these materials decrease rapidly with increasing calcination temperature. Further studies are needed to correlate the catalytic properties of these substances with accurate measurements of the active species in order to optimize the chemistry of these important materials.

#### **FEASIBILITY STUDIES FOR ON-LINE RECYCLING ALUMINUM ALLOY SORTING USING PROMPT GAMMA-RAY ACTIVATION ANALYSIS**

A major aluminum company is committed to a recycling program for aluminum scraps from automobile parts. For this reason, NIST scientists, in conjunction with scientists at North Carolina State University (NCSU) and the company are working to develop a method for rapid nondestructive identification of aluminum alloys that can be used

on-line. Prompt gamma-ray activation analysis (PGAA) appears to be an excellent choice for such a method. The technique uses a beam of neutrons to stimulate emission of gamma-radiation in samples. PGAA gives adequate detection limits for measurement of key elements (magnesium, aluminum, manganese, iron, copper, zinc) used for identifying alloys. Furthermore, since the neutron and gamma radiation penetrate the sample, the presence of surface coatings of paint, grease, or dirt does not significantly affect the identification.

Preliminary measurements, performed using PGAA instruments at the NCSU PULSTAR reactor and at the NIST Cold Neutron Research Facility, indicate that this approach to aluminum alloy sorting is possible. Although both instruments utilize neutrons produced in the core of a reactor, the NIST facility uses only low energy "cold" neutrons in the analysis, yielding lower background and better sensitivities for most elements. The NIST instrument has given reliable measurement of Mn, Fe, Cu, and Zn at mass fractions of <2 % in 0.2 g to 1.3 g samples of aluminum alloys. Despite the promise shown by these preliminary studies, the ultimate success of the method depends upon implementing a neutron source that can be used on-line. Future studies will be devoted to developing a portable  $^{252}\text{Cf}$  source that may be used in conjunction with a cold moderator to produce low energy neutrons.

#### **U.S./CANADA/MEXICO QUALITY ASSURANCE (QA) PROGRAM FOR AQUATIC AND ATMOSPHERIC ENVIRONMENTAL CHEMICAL MEASUREMENTS**

On Nov. 15–17, 1994, 29 experts in environmental chemical measurements and related QA issues met in Queretaro, Mexico, to discuss the development of a chemical measurements QA program for the NAFTA countries. The planning meeting was organized and spearheaded by chemical measurements QA experts from NIST and the National Oceanic and Atmospheric Administration (USA), National Research Council (Canada), and the Centro Nacional de Metrologia (Mexico). A representative from QUASIMEME, a European QA program for marine analytical measurements, was invited to attend to present the European perspective.

The collective vision of those attending was to establish a QA program to assure that environmental information produced by the NAFTA countries is compatible and of known quality, thereby providing a reliable and harmonized characterization of

the North American environment. It was mutually agreed that the mission for the North American Chemical Measurements Quality Assurance Program would be to clarify scientific understanding of the North American environment, promote comparability of data produced for the three NAFTA countries, and provide tools for quality decision making.

The U.S. Department of Commerce involvement in the proposed chemical measurements QA program would be coordinated through NIST and NOAA, with NIST responsible for the overall U.S. leadership and coordination.

#### **WORKSHOP HELD ON THE CHEMICAL ANALYSIS OF AUTO CATALYSTS**

On Dec. 1–2, 1994, NIST held a workshop on the determination of platinum group elements in automobile catalysts using isotope dilution and inductively coupled plasma mass spectrometry (ID-ICP-MS). This workshop was arranged as part of a cooperative research and development agreement with a private company and brought together analysts from the company, an automotive company, and the University of Arizona. The goal of the workshop was to transfer NIST methodology to the participants; the detailed procedures for sample preparation and instrumental measurement used at NIST to achieve high-accuracy chemical analysis of automobile catalysts were demonstrated by research chemists.

The economic value of the platinum, palladium, and rhodium incorporated in auto catalysts is substantial, \$0.5 billion to \$1 billion per year, and their analysis is notoriously difficult. This project is an extension of work done to certify Standard Reference Materials (SRMs) 2556 and 2557, Used Auto Catalysts, which were issued in 1993. These standards were developed at the request of the International Precious Metals Institute to provide an accuracy base for comparison of the precious metal assays. The transfer of the methodology of ID-ICP-MS used to certify the SRMs directly to laboratories will provide them not only the accuracy required but also the precision for careful accounting of the precious metal inventories. The participants will be demonstrating their success in applying ID-ICP-MS in the next phase of the collaboration.

#### **EBIT DISCOVERS "FORBIDDEN" LIGHT**

NIST scientists working with the new electron beam ion trap (EBIT) have detected and measured

visible light emitted from barium and xenon atoms that have been stripped of many of their electrons. Such highly ionized matter typically radiates x rays but also can produce significant amounts of visible light due to transitions "forbidden" by lowest order quantum mechanical selection rules. This visible light can be of great benefit to researchers trying to develop advanced technological devices that use highly charged matter, such as Tokamak plasma fusion reactors that hold promise as a plentiful source of electrical power. Light from similar "forbidden" transitions has been used for a number of years as a plasma diagnostic tool in Tokamaks around the world, but wavelengths of all previously known forbidden lines quickly shift into the vacuum ultraviolet as the plasma temperature is raised. Thus the advantage of remote observations with optical fibers is lost, and the line widths decrease strongly, making them less suitable for ion temperature measurements.

Three years ago, scientists from NIST and NRL predicted that a particularly unusual sequence of forbidden lines should exist in heavy elements having all but 22 electrons removed. These titanium-like ions of elements in the entire upper half of the periodic table were predicted to emit visible or near-ultraviolet radiation for a wide range of plasma temperatures. The recent work on the NIST EBIT provides the first experimental look at these predicted spectral lines. After measuring their exact wavelengths, NIST and NRL researchers initiated work with scientists from a private laboratory to use high-resolution Fabry-Perot interferometry to infer information about local plasma temperature and magnetic fields inside the EBIT by measuring Doppler and Zeeman broadening of the lines. Plans are now in progress to detect this light directly on a Tokamak fusion device.

#### **MULTIAGENCY SOLAR ULTRAVIOLET INTERCOMPARISON HELD**

An interagency ultraviolet intercomparison was held Sept. 19–29, 1994, to assess the performance of spectroradiometers used in solar ultraviolet monitoring networks. The primary focus of the intercomparison was to synchronize solar ultraviolet irradiance measurements over 6 days, from mid-morning to sunset. Organized by NIST and NOAA's Environmental Research Laboratory, this intercomparison was conducted on Table Mountain, a mesa approximately 16 km north of Boulder, CO. Participating agencies were the

Department of Agriculture, the Environmental Protection Agency, the National Science Foundation, the Smithsonian Environmental Research Center, and the Atmospheric Environmental Service of Canada. The instruments were characterized indoors for wavelength accuracy using a Hg lamp, for slit-scattering function and stray-light rejection using a HeCd laser, and for irradiance calibration using a NIST irradiance standard lamp.

### **COUNCIL ON IONIZING RADIATION MEASUREMENTS AND STANDARDS MEETS AT NIST**

The Council on Ionizing Radiation Measurements and Standards (CIRMS) held its third annual meeting at NIST Nov. 16–18, 1994. The organization represents thousands of users of ionizing radiation and radioactive sources engaged in industrial radiation processing and sterilization, medical radiation diagnostics and therapy, nuclear power, and worker radiation protection programs. CIRMS provides a forum for discussing ionizing radiation issues; identifying, defining and prioritizing needed work; disseminating information on standards; and organizing workshops and meetings to advance ionizing radiation technology.

Over 100 participants discussed measurement quality assurance for the different ionizing radiation communities. Representatives attended from 33 corporations, nine federal agencies, seven national laboratories, seven universities, and one state agency. The first day was devoted to activities of the four subcommittees of the Science and Technology Committee: Radiation Effects, Medical Technology, Environmental Technology, and Occupational Radiation Protection. In the plenary session, invited talks were presented in the following areas: basic concepts of measurement quality assurance; recent developments from NIST's National Voluntary Laboratory Accreditation Program (NVLAP), the FDA, and states; standards and techniques for uncertainty analysis; characterization and traceability of radioactive sources; and comparability of measurement quality assurance programs in different fields. The participation was particularly high from secondary calibration laboratories and from radiation instrument manufacturers, as CIRMS provides a forum for these groups to discuss directions for new programs and instrument needs.

### **MAGNETORESISTANCE EXCEEDS 20 % IN SYMMETRIC SPIN VALVES**

Over the next few years, a new generation of ultra-high-density computer disk drives will hit the market. These drives will use thin-film magnetic multilayers, known as symmetric spin valves, as magnetic sensors (read heads) to read the magnetic bits on the disk.

Advanced Technology Program (ATP) funding is supporting the development of these read heads through a grant to the National Storage Industry Consortium (NSIC), which consists largely of the U.S. hard disk drive industry. Research in support of NSIC carried out at NIST has developed a symmetric spin valve with a magnetoresistance of 21.2 %, the highest value ever recorded for a symmetric spin valve. (The largest previously reported value is 14 %.) The magnetoresistance is the change in the electrical resistance of the film in response to the changing magnetic field direction as the read head flies over the bits on the disk. Thus, the magnetoresistance is a measure of the sensitivity of the read head; the more sensitive it is, the more densely bits can be stored on and read from a disk. The NSIC goal is 1.5 Gbit/cm<sup>2</sup>. The NIST research constitutes an important step toward achieving that goal. This program recently has transferred to join a complementary program in support of NSIC already located at NIST.

### **FUTURE NEEDS IN ELECTRONIC PACKAGING AND INTERCONNECTION**

The results of a recent industry-university-government workshop on "Materials Metrology and Data for Commercial Electrical and Optical Packaging and Interconnection Technologies," conducted May 5–6, 1994, are given in NISTIR 5520, *Metrology and Data for Microelectronic Packaging and Interconnection*.

The report identifies cross-cutting barriers, critical technical challenges, and opportunities for NIST in metrology and data for electronic packaging and interconnection technologies. Specific barriers that apply to all product applications considered and all technical challenges identified include *in-situ* metrology for manufacture and in-use; materials' data and databases; modeling, design, and manufacturing tools; and a technology infrastructure. Technical challenges viewed to strongly impact the performance and reliability of

materials used in electrical and optical packaging and interconnection include adhesion, moisture measurement and control, micro thermo-mechanical measurements, dimensional stability, failure mechanisms, high frequency electrical measurements, and interconnection.

Industry believes NIST could help meet their needs by:

- spearheading the establishment of a national task force on packaging and interconnection metrology and data that brings together key segments of the microelectronics industry, facilitates advancements, coordinates research and disseminates results;
- conducting with industry an objective assessment of existing industry test standards, new standard needs, and current materials' databases;
- developing fundamental understanding of materials and material structures;
- developing and disseminating improved measurement and testing methods for materials and material structures, especially techniques well suited for the manufacturing environment;
- broadly disseminating R&D expertise and results to industry by employing round robin testing; and
- developing a standards program that seeks adoption of improved methods by national and international standards bodies, producing Standard Reference Materials and material structures, and developing and testing standards for the electronic exchange of materials' data.

The May 1994 workshop attracted more than 100 technical experts from leading U.S. companies in advanced materials, semiconductor, and printed wiring board manufacturing and assembly as well as interested representatives from academia and government. The intent of the workshop was to address the challenges and priorities in materials' measurement and data to support the design, manufacture, and reliability assessment of critical electrical and optical packaging and interconnection materials, structures, and processes, and to build upon recent industry planning conducted by the Semiconductor Industry Association, the Institute for Interconnecting and Packaging Electronic Circuits (IPC), and the Optoelectronics Industry Development Association (OIDA).

## NIH/NIST BIOMOLECULAR DYNAMICS WORKSHOP

On Dec. 3, 1994, NIST and the National Institutes of Health held a joint workshop on the potential applications of neutron spectroscopy to biomolecular dynamics. The dynamical behavior of biological macromolecules and associated water is often a key factor in determining biological function or biological processes. The workshop was attended by approximately 55 scientists from government laboratories, academia, and industry. Five invited speakers from NIH and the Food and Drug Administration described their research efforts and elucidated specific questions that might be answered by performing complementary inelastic neutron scattering experiments.

With the addition of three state-of-the-art inelastic neutron scattering instruments, due to become operational within the next 18 months, NIST's Cold Neutron Research Facility (CNRF) will offer a capability, unique in the United States, to *directly* probe a wide range of dynamic processes encompassing the  $10^{-7}$  s to  $10^{-12}$  s time scales. Thus, processes such as macromolecular and water diffusion and segment flexibility can be measured in addition to high-frequency intramolecular motions and diffusive and vibrational modes. The measured scattering cross-sections relate to amplitudes as well as frequencies of motion and can be compared directly with those calculated from molecular dynamics computer simulations. A key to these experiments is the possible substitution of deuterium for hydrogen in the macromolecules or the water in order to better isolate the dynamics of molecular components of interest.

## FIRST CD-ROM OF NIST FIRE PUBLICATIONS

NIST has created the first CD-ROM as an information resource for use in the fire community. This CD-ROM, Fire Research Publications, 1993, contains the complete contents of the 1993 works published by the fire research staff and related grant reports. Users can print from their own personal computers the complete reports or selected pages from approximately 5000 pages of text, graphics, and photographs that are imaged on the disk. Initial reactions from individuals and libraries have been very favorable. The disk is seen as a cost-effective means of distributing and storing technical information.

## **SECOND YEAR RESULTS FOR STUDY OF FIRE DETECTOR SITING FOR COMPLEX CEILINGS**

NIST has released a report on the results of the second year of phase 2 of the International Fire Detection Research Project. This four-year effort is funded by a consortium of federal agencies, private companies, and industry associations through the National Fire Protection Research Foundation. The objective of the project is to establish optimum siting requirements for fire sensors located in rooms with complex ceiling geometries and high ventilation rates. This will be accomplished through the use of state-of-the-art computational fluid dynamics modeling of the distribution of temperature, velocities, and particulates in these spaces as a function of compartment geometry and fire size.

In the second year, this study examined the details of the fire driven flows moving up sloped ceilings over a range of slopes that may be encountered in buildings. These ceilings included exposed beams running both in the direction of, and across, the slope and further included the effects of gaps between the beam and ceiling surface. The results were used to determine optimum siting guidelines for fire sensors that could be incorporated directly into standards.

This study is being heralded by the alarm industry as providing the first insights into aspects of fire sensor performance that have been incapable of study experimentally. Revised installation guidelines based on these results also may reduce the costs of providing fire protection in commercial properties in which such ceiling arrangements are found.

## **NIST SUPPORTS ACI LABORATORY TECHNICIAN CERTIFICATION PROGRAM**

NIST is continuing to expand its programs to promote the quality of testing in construction materials laboratories. A memorandum of agreement between NIST, ASTM, and the American Concrete Institute (ACI) calls for NIST to provide performance examinations for the Concrete Technician Laboratory Certification Program of the ACI. The performance examination is available to technicians in the almost 300 concrete and aggregate laboratories currently inspected by NIST. ACI provides the written examination and the formal certification of technicians who successfully pass both examinations. As of November 1994, 18 technicians have taken the NIST performance examination.

## **SECRETARY OF COMMERCE APPROVES FEDERAL INFORMATION PROCESSING STANDARD (FIPS) FOR THE GOVERNMENT INFORMATION LOCATOR SERVICE**

Secretary Brown recently approved FIPS 192, Application Profiles for the Government Information Locator Service (GILS), to create a uniform technical approach to providing information locator services to the public. Based on a voluntary industry standard, the American National Standard for Information Retrieval, Application Service Definition and Protocol Specification for Open Systems Interconnection (ANSI/NISO Z39.50), the application profile was adapted for government systems by a group of industry and government experts led by the U.S. Geological Survey.

FIPS 192 facilitates the implementation of GILS, a decentralized collection of computer servers and associated information services that the public can use to find information throughout the federal government. The profile addresses some of the interactions between computer systems, allowing users of client software to interconnect with GILS-compliant servers containing accessible public information. The profile is expected to be implemented in commercial, off-the-shelf products that will improve information retrieval for users and expand choices for federal agencies.

## **NEW PUBLICATION FOCUSES ON ELECTRONIC COMMERCE**

NIST Special Publication 500-218, Analyzing Electronic Commerce, presents an overview of electronic commerce and examines key issues involved in its deployment. After looking at ways in which electronic commerce is presently being conducted, most notably with electronic data interchange (EDI), the report defines an architectural model for needed supporting services, such as the user interface, data communications, data management, and security. The report predicts the significant expansion of electronic commerce applications within the federal government and U.S. industry over the next decade and concludes that communications, data management, and security modules are essential for establishing an electronic commerce infrastructure.

## **NIST INITIAL GRAPHICS EXCHANGE SPECIFICATION (IGES) TEST SERVICE**

NIST initiated the IGES Test Service to evaluate conformance of IGES processors to FIPS 177, IGES, and the Continuous Acquisition and Life-

Cycle Support (CALS) specification MIL-D-28000, Class II subset (Engineering Drawings). Both FIPS 177 and MIL-D-28000 adopt the American National Standard Digital Representation for Communication of Product Definition Data, ASME/ANSI Y14.26M-1989, the equivalent of IGES Version 4.0. IGES specifies file structure and syntactical definition and defines the representation of geometric, topological, and nongeometric product definition data.

Conformance testing of IGES processors maximizes the probability of successful data exchange between dissimilar computer-aided design and computer-aided manufacturing (CAD/CAM) systems, and ensures that the basic concepts and geometry of IGES are implemented correctly. The testing methodology used determines whether a given preprocessor or postprocessor is able to create or read conforming data files. Emphasis is placed on single entity testing, considered the basic building block for more complex structures. In addition, testing for conformance to the IGES 5.1 Specification is also available upon request.

#### **INTERNET FIREWALLS THE SUBJECT OF NEW PUBLICATION**

NIST Special Publication 800-10, Keeping Your Site Comfortably Secure: An Introduction to Internet Firewalls, provides an overview of the Internet and security-related problems. It describes firewall components, the reasoning behind firewall usage, several types of network access policies, and resources for more information. The document assists federal and industry users in planning and purchasing a firewall.

#### **TESTS OF MAGNETIC STORAGE MATERIALS SHOW PROMISE**

One of the major goals of the computer industry is to pack more and more bits and bytes into magnetic storage systems. A recent collaborative effort between NIST and a private company shows promise for an advanced magnetic read head structure. Tests indicated that magnetic materials made from nickel-iron/silver multilayer thin films exhibit low magnetostriction (the tendency of materials to expand or contract when a magnetic field is applied) as well as impressive sensitivity in low magnetic fields. Improving the sensitivity of the magneto-resistive heads used to read the magnetic fields of the storage medium is critical to increasing storage density. However, the head should not be so sensitive to other physical parameters that it is difficult to manufacture or unreliable to use. Mag-

netostriction must be maintained very close to zero. To test the performance of nickel-iron/silver systems, a NIST scientist deposited the thin films on silicon wafers. Following dicing and annealing, the magnetoresistance of the resulting specimens was measured at NIST and the magnetostriction was measured at the private company. Annealing is necessary for the nickel-iron/silver multilayers to exhibit giant magnetoresistance. The researchers were able to identify an optimal annealing temperature, which results in zero magnetostriction and high sensitivity in low magnetic fields. For further information, contact Steve Sanders at (303) 497-5096 or Young Kim at (303) 673-6125.

#### **REPORT SAYS FEDERAL PROGRAM HELPING INVENTORS**

By the end of 1992, an 18 year-old Federal program sponsored jointly by NIST and the Department of Energy had helped individual inventors and small companies develop and market at least 129 ideas for saving energy. These new technologies have generated sales of \$763 million and earned more than \$18 million in royalties for inventors who licensed their inventions, says a recent report by the Oak Ridge National Laboratory. As part of the Energy-Related Inventions Program, NIST provides free technical evaluations of inventions and recommends those that have commercial promise to DOE for possible financial support. The program also helps inventors find other support such as market assessments, concept or prototype development, and laboratory testing. The Oak Ridge report also said that 36 spinoff technologies generated an additional \$63 million in sales. Since it was not feasible to assess the energy saved by all of the ERIP-supported technologies that entered the market, the Oak Ridge evaluators examined three of the biggest sellers. These three inventions saved more than \$531 million in energy expenditures over the past decade resulting in reduced emissions of nearly 1 million metric tons of carbon. With \$41 million in grants awarded from 1975 through 1992, and \$106 million in program appropriations over the same period, ERIP has generated a 19 to 1 return in terms of sales to grants, and a 7 to 1 return in sales versus program appropriations. The Economic, Energy, and Environmental Impacts of the Energy-Related Inventions Program (ORNL/CON-381) is available for \$19.50 prepaid from the National Technical Information Service, Springfield, VA 22161, (703) 487-4650. Order by DE 94-017162.



### **UNITED STATES, INDIA AGREE TO MUTUALLY RECOGNIZE LABS**

To enhance trade between the United States and India, a letter of intent was signed recently for the development of a Mutual Recognition Agreement (MRA) for laboratory accreditation between the National Accreditation Board for Testing and Calibration Laboratories, Department of Science and Technology, India, and the U.S. National Voluntary Laboratory Accreditation Program (NVLAP) at NIST. International guidelines for mutual recognition agreements will be the guiding basis for the new MRA. Both institutions have announced conformance to international guidelines for laboratory accreditation programs. The achievement of an MRA promotes common interest in assisting commerce and encourages specific laboratory interests in providing demonstrated competent performance testing and calibration services. For information, contact Lawrence Galowin, NVLAP, A162 Building 411, NIST, Gaithersburg, MD 20899-0001, (301) 975-4016, fax: (301) 926-2884.

### **BARRIERS TO NII HIGHLIGHTED IN NEW REPORT**

A basic resistance to change will keep people and organizations from taking advantage of the information infrastructure, according to a new report, *Breaking the Barriers to the National Information Infrastructure (NII)*. The report, which highlights events at a September 1994 conference in Washington, DC, summarizes panel discussions on manufacturing, education, electronic commerce, health care, and entertainment. In addition to the findings, the report reviews 28 demonstrations of various technology applications that were part of the 2 day conference. The conference was co-sponsored by the Council on Competitiveness and the Clinton Administration's Information Infrastructure Task Force. For a copy of the report, which costs \$25 plus \$3 for shipping and handling, call the Council on Competitiveness at (202) 682-4292. The report is also available electronically by gopher: telnet (login = gopher) or anonymous ftp to iitf.doc.gov, and on the World Wide Web at <http://iitf.doc.gov>. For technical assistance when accessing the report electronically, contact Carol Barnes at (301) 975-3243.

### **WEIGHTS AND MEASURES DEVICES HANDBOOK UPDATED**

NIST Handbook 44-1995, *Specifications, Tolerances, and Other Technical Requirements for Weighing and Measuring Devices*, was updated at the 79th Annual Meeting of the National Conference on Weights and Measures in July 1994. Among the changes is a new definition for the concentrated load capacity of vehicle, axle-load, and livestock scales. A tentative code for near-infrared grain analyzers has been added for experimental use and comment. The tentative code will be applied to devices submitted to NCWM's National Type Evaluation Program but will not be enforced by weights and measures officials. The Mass Flow Meters Code, previously tentative, is now permanent. Changes also have been made to accommodate a new method of sale for compressed natural gas used as an engine fuel. For information, contact the Office of Weights and Measures, A357 Physics Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-4004. NIST Handbook 44 is available for \$16 prepaid from the U.S. Government Printing Office, Washington, DC 20402. Order by stock no. 003-003-03298-1.

### **NEW PROGRAM GOAL: HELP MANUFACTURERS WORK CLEANER**

NIST's Manufacturing Extension Partnership (MEP), in cooperation with the Environmental Protection Agency, is seeking project proposals from non-profit organizations that, if funded, will help the nation's 370,000 smaller manufacturers become environmentally sound while remaining competitive. The jointly funded competition is expected to award more than \$3 million in cooperative agreements and financial assistance for selected projects in three areas: integrating environmentally focused technical assistance into the services of the MEP's national extension center network; developing environmentally related technical assistance tools and techniques; and implementing pilot information centers to provide specific industries with access to data on innovative technologies, pollution prevention opportunities, and regulatory compliance. Solicitations for proposals will be announced in the *Commerce Business Daily*. For more information, contact David Gold, MEP Environmental Projects Manager, B115 Polymer Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-5049,

## REPORT DETAILS NIST WORK IN SUPERCRITICAL FLUIDS

In recent years there has been renewed interest in the use of supercritical extraction for industrial chemical separation processes. In keeping with its mission to perform research that benefits U.S. industry, NIST maintains an active program in supercritical fluid technology and closely related fields. A new paper explains the current research in these areas under way at NIST. For example, NIST is evaluating fluorinated alternative refrigerants and other fluorinated hydrocarbons for use as solvents in liquid and supercritical fluid extraction processes. These fluids are important as alternative solvents to chlorinated species that may contribute to ozone depletion. In another case, the solubility of pharmaceuticals in alternative refrigerants and carbon dioxide/micelle systems is being studied for drugs that are not sufficiently soluble in supercritical fluid carbon dioxide. Work with alternative refrigerants also has included chemical and thermophysical characterization of the fluids themselves, including the measurement and modeling of fundamental thermodynamic and transport properties, and the development of a compendium of analytical data. For copies of paper 51-94, contact Sarabeth Moynihan, Div. 104, NIST, Boulder, CO 80303-3328, (303) 497-7765, e-mail: moynihan@bldrdoc.gov (via Internet).

## HANDBOOK FOR ALTERNATIVE REFRIGERANTS AVAILABLE

A new handbook edited by a NIST researcher will be of interest to laboratories developing and using new fluids as refrigerants and solvents to replace the fully halogenated materials believed to contribute to atmospheric ozone depletion. The 700-page volume, which is divided into two sections, provides a protocol for the chemical analysis of new fluids that are synthesized, prepared, and tested. It also provides a compendium of data to implement the protocol. In the first section, all the major chemical analysis methods developed and used at NIST and other laboratories are reviewed, including spectroscopic, chromatographic, and "wet" chemical analysis. The treatment is divided into three main parts: qualitative identification, quantitative determinations, and chemical reaction screening. The second section contains a compilation of analytical information on the new fluids and their products. This section provides (for each fluid or product) physical properties, health and safety information, mass spectra, infrared spectra, ultra-

violet spectra, nuclear magnetic resonance spectra, and gas chromatographic retention data. For more information on the Handbook for the Analysis and Identification of Alternative Refrigerants, contact Tom Bruno at Div. 838, NIST, Boulder, Colo. 80303-3328, (303) 497-5158.

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## Standard Reference Materials

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### OXYGENATES IN GASOLINE STANDARD REFERENCE MATERIALS (SRMs)

NIST has just issued a new series of Standard Reference Materials, Oxygenates in Gasoline, which respond to the measurement needs included in legislation enacted in 1990 by the Federal Government and the state of California requiring a significant reduction in automobile exhaust emissions. Many studies have shown that the exhaust emissions from automobiles are directly related to the composition of the gasoline used. Thus a major pathway to the mandated reduction of emissions is by reformulation of gasoline. The addition of oxygenated hydrocarbons, usually in the form of aliphatic ethers, can significantly reduce hydrocarbon and carbon monoxide emissions. For example, it has been shown that the addition of 2.7 mass % oxygen to commercial unleaded gasoline can result in the lowering of the hydrocarbon and carbon monoxide emissions by 6 % and 11 %, respectively.

Federal and California regulations will shortly require that all commercial gasoline contain 2.7 and 2.0 mass % oxygen, respectively. To meet these oxygen content requirements, refiners will be blending aliphatic ethers and/or ethanol into unleaded gasoline feed stock at levels of between 10 to 15 mass %. A number of single ethers, ether mixtures and alcohols probably will be employed, the most common materials being methyl t-butyl ether (MTBE), ethyl t-butyl ether (ETBE), t-amyl methyl ether (TAME), and ethanol. Analytical methods for the determination of ethers within a complex hydrocarbon mixture are not trivial and require calibration through the use of accurate chemical standards. The California Air Resources Board (CARB), Western States Petroleum Association (WSPA), ASTM Committee D-2, and the Energy Resources Council (ERC) have requested that NIST undertake the development of suitable SRMs.

Eight oxygenate in gasoline SRMs have been prepared and certified at NIST. Available now are four SRMs containing ethanol, MTBE, ETBE, or TAME in reference gasoline at an oxygen concentration of 2.0 %. Also available are three SRMs containing MTBE, ETBE, or TAME in reference gasoline at an oxygen concentration of 2.7 %. An SRM containing ethanol in reference gasoline at an oxygen concentration of 3.5 % is also available. In addition to these SRMs, eight NIST Traceable Reference Materials (NTRM) with identical concentrations and composition are available commercially. The NTRM program is a new NIST service that allows any commercial supplier to produce and sell NIST traceable materials that closely match available SRMs. NIST value assigns all NTRMs based on SRMs currently being sold. The NTRM program currently includes only gas SRMs and these new oxygenate in gasoline materials.

#### **STANDARD REFERENCE MATERIAL 1511 – MULTI-DRUGS OF ABUSE IN FREEZE-DRIED URINE**

Drug testing programs are in wide use today in employment situations. The programs seek to identify illegal drug usage that adversely affects workplace safety and causes a dramatic decrease in employee productivity. It is paramount that any testing program base its results on accuracy both in terms of user identification and to prevent false positives. To accomplish the required accuracy, drug testing laboratories must rigorously follow a quality assurance plan that includes quality control of screening procedures and confirmatory analytical methods.

In response to requests from drug testing laboratories citing the need for accurate and NIST-traceable standards, NIST announces the development and certification of SRM 1511—Multi-Drugs of Abuse in Freeze-Dried Urine. SRM 1511 consists of three bottles of freeze-dried urine, each containing confirmatory test cutoff levels of morphine, codeine, cocaine metabolite (benzoylecgonine), marijuana metabolite (THC-9-COOH), and phenylclidine. The concentrations of the drugs in SRM 1511 meet cutoff levels as established in guidelines by the National Institute on Drug Abuse. With the availability of SRM 1511, laboratories performing drug testing now have a NIST SRM with known concentrations of target drugs to test the accuracy of their analyses.

The current availability of other drug abuse SRMs includes SRM 1507b—THC-COOH in Urine, SRM 1508—Cocaine & Metabolites in Urine, SRM 2381—Morphine-Codeine in Urine, SRM 2382—Morphine-Glucuronide in Urine, RM 8448—Drugs of Abuse in Hair Segments, and RM 8449—Drugs of Abuse in Hair.

#### **STANDARD MAKES SPINACH MORE PALATABLE TO INDUSTRY**

NIST chemists have just issued a fourth botanical Standard Reference Material, Spinach Leaves, to help agriculturalists and food producers ensure accurate analyses. The Spinach Leaf SRM joins botanical SRMs of apple, peach, and tomato leaves, which all help food growers and producers accurately measure nutritional and toxic elements in their products. Scientists in the food industry will use the SRM to assess whether their own analytical methods are giving accurate results. NIST chemists have certified the concentration of 19 elements in the finely powdered, freeze-dried leaves. Certified values are given for nutrients, such as calcium, potassium, and zinc, and toxic elements, such as arsenic and mercury. SRM 1570a is available for \$257 from the NIST SRM Program, Building 202, Room 204, Gaithersburg, MD 20899, (301) 975-6776, fax: (301) 948-3730.

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# Evaluating and Expressing the Uncertainty of Measurement Results



Uncertain about expressing measurement uncertainty? Do you need to know how NIST states the uncertainty of its measurement results and how you can implement their internationally accepted method in your own laboratory? Then you need the newly available 1994 edition of the National Institute of Standards and Technology Technical Note 1297, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*.

The 1994 edition of the National Institute of Standards and Technology Technical Note 1297, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, by Barry N. Taylor and Chris E. Kuyatt is now available.

The 1994 edition of TN 1297 includes a new appendix—Appendix D—which clarifies and gives additional guidance on a number of topics related to measurement uncertainty, including the use of certain terms such as accuracy and precision. Very minor word changes have also been made in a few portions of the text of the 1993 edition in order to recognize the official publication in October 1993 by the International Organization for Standardization (ISO) of the *Guide to the Expression of Uncertainty in Measurement* on which TN 1297 is based. However, the NIST policy on measurement uncertainty, Statements of Uncertainty Associated with Measurement Results, which is reproduced as Appendix C of TN 1297, is unchanged.

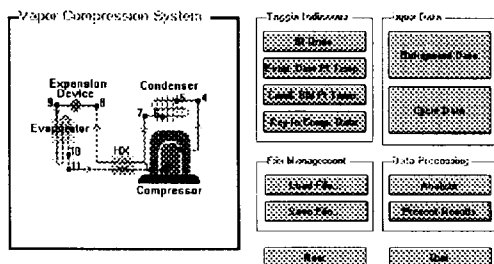
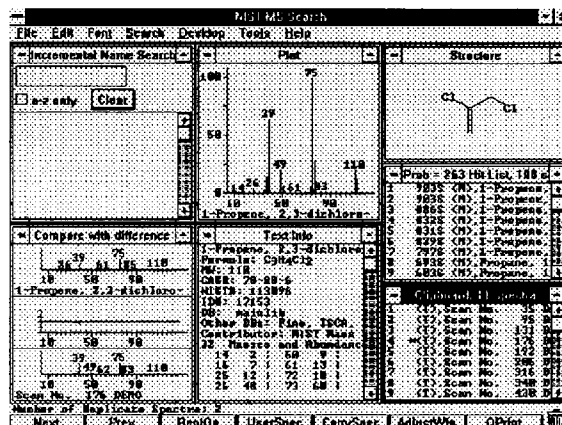
It is expected that the 1994 edition of TN 1297 will be even more useful than its immediate predecessor, the 1993 edition, of which 10 000 copies were distributed worldwide.

Those United States readers who wish to delve into the subject of measurement uncertainty in greater depth may purchase a copy of the 100-page ISO *Guide* from the Sales Department of the American National Standards Institute (ANSI), 105-111 South State Street, Hackensack, NJ 07601. Copies may also be purchased from the ISO Central Secretariat, 1 rue de Varembé, Case postale 56, CH-1211 Genève 20, Switzerland.

Single copies of the 20-page TN 1297 may be obtained from the NIST Calibration Program, Building 820, Room 232, Gaithersburg, MD 20899-0001, telephone: 301-975-2002, fax: 301-948-3825.

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## *Periodical*

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## *Nonperiodicals*

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**Monographs**—Major contributions to the technical literature on various subjects related to the Institute's scientific and technical activities.

**Handbooks**—Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

**Special Publications**—Include proceedings of conferences sponsored by NIST, NIST annual reports, and other special publications appropriate to this grouping such as wall charts, pocket cards, and bibliographies.

**National Standard Reference Data Series**—Provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated. Developed under a worldwide program coordinated by NIST under the authority of the National Standard Data Act (Public Law 90-396). NOTE: The Journal of Physical and Chemical Reference Data (JPCRD) is published bimonthly for NIST by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements are available from ACS, 1155 Sixteenth St., NW, Washington, DC 20056.

**Building Science Series**—Disseminates technical information developed at the Institute on building materials, components, systems, and whole structures. The series presents research results, test methods, and performance criteria related to the structural and environmental functions and the durability and safety characteristics of building elements and systems.

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**NIST Interagency Reports (NISTIR)**—A special series of interim or final reports on work performed by NIST for outside sponsors (both government and nongovernment). In general, initial distribution is handled by the sponsor; public distribution is by the National Technical Information Service, Springfield, VA 22161, in paper copy or microfiche form.

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